AMENDED SPECIFICATION

Reprinted as amended in accordance with the Decision of the Superintending Examiner acting for the Comptroller General, dated the sixteenth day of November, 1966, under Section 33, of the Patents Act, 1949.

PATENT SPECIFICATION

949,345

NO DRAWINGS

949,345

Inventors: PETER WILLIAM YEOMANS and JOHN WILSON REDMAN

Date of filing Complete Specification: Dec. 3, 1962.
Application Date: Dec. 20, 1961. No. 45671/61.
Complete Specification Published: Feb. 12, 1964.

Index at acceptance:—C1 B(3C2, 3C3, 3C4, 3F1)
International Classification:—C05b, d, f.

COMPLETE SPECIFICATION

Improved method of producing Granular Fertilisers

We, LAWES CHEMICAL COMPANY LIMITED of Creeksmouth, Barking, Essex, EDWARD WEBB & SONS (STOURBRIDGE) LIMITED of Wordsley, Stourbridge, Worcestershire, and ANDERTON - RICHARDSON FERTILISERS LIMITED, of Skeldergate Bridge, York, Companies registered under the Laws of Great Britain, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the preparation of granular fertilisers containing monocalcium phosphate and urea, and in particular to the preparation of granular fertilisers containing monocalcium phosphate and a high proportion of urea, for example, sufficient urea to give from 15% and upwards by weight of urea in the finished product.

When urea is incorporated into fertilisers containing monocalcium phosphate there are no serious problems involved in granulating or drying the fertiliser in conventional equipment, provided that the content of urea is kept below 12% to 15% by weight of the finished product.

If however, it is desired to use more than 15% by weight of urea in the fertiliser then technical problems become evident during the drying and later stages of the process which render continuous production inoperable under conditions normally applicable in fertiliser granulation.

These problems arise during processing of the fertiliser because of the reaction of urea with the other constituents of the fertiliser, for example, with the monocalcium phosphate constituent and any uncombined free phosphoric or sulphuric acid contained in the fertiliser and with ammonium sulphate and potassium chloride and potassium sulphate if they are present.

The chief difficulty in processing relates to the operation of the rotary dryer wherein the fertiliser granules rapidly assume a wet sticky condition and adhere tenaciously to each other and to the interior surface of the dryer and any mechanical lifting flights. This build-up of sticky material within the dryer occurs very quickly and is so extensive that the dryer has to be stopped and cleaned after one or two hours working. In addition due to the sticky nature of the material emerging from the dryer there is a continual likelihood of stoppages in other parts of the plant caused by blockages and sticky build-up, for example, at the screens and in the chutes and on the conveying equipment.

We have found that the difficulties caused by sticky build-up which arise during the production and especially the drying, in conventional equipment, of granular fertilisers containing free uncombined acids and monocalcium phosphate and a high proportion of urea, for example, 15% or more of urea by weight of the finished product, may be avoided by reducing the amount of any uncombined free acid that is present in the fertiliser granules before the granules are passed to the drying stage of the process. Such a neutralising step also has advantages with lower urea concentration. The amount of free acid to be neutralised depends on the type and formulation of the fertiliser and also on the quantities of urea and monocalcium phosphate containing constituents contained in the fertiliser.

The invention consists in a method for reducing dryer stick in the preparation of granular fertilizers, which comprises carrying out the preparation in the three separate and consecutive steps (i) preparing a mixture containing monocalcium phosphate, urea, and free uncombined acid and granulating the mixture (ii) after granulation applying a neutralising agent to the formed granules to reduce the overall acid content and (iii) drying the granules.

In general it is sufficient to reduce the free uncombined acid in the monocalcium phosphate constituent to about 1% by weight of the monocalcium phosphate-containing constituent present.

If in addition to the free acid contained in the monocalcium phosphate containing constituent there is also free acid present from other sources, such as that contained in the other fertiliser materials used in the formulation or added deliberately, such as sulphuric and/or phosphoric acids, added before or during granulation (and in any case before addition of neutralising agent) either to promote granulation or to provide nutrient value, then it is necessary that before the granulated mixture is passed to the drying stage sufficient neutralising agent or agents are added to convert any and all such deliberately added free acids so present to their salt or salts. This amount of neutralising agent is additional to that required to reduce the free acid content of the monocalcium phosphate containing constituent to its desired level.

Preferably, the mixture contains ordinary super phosphate or triple super phosphate (which, as commercially available, generally contain from 3 to 6% of free uncombined acid) and urea. It is also preferred that at least 15% by weight of urea should be present, while the urea concentration may be up to 70% by weight.

In the drying stage of the process the granules are in general dried to a temperature less than 95° C and preferably between 80° and 90° C with subsequent cooling.

The neutralising agent may be gaseous, liquid or solid, and suitable neutralising agents are, e.g. gaseous ammonia, aqueous ammonia solution, aqueous sodium or potassium hydroxide solution, solid hydrated lime, finely divided calcium carbonate, and finely divided solid diammonium phosphate.

In any case the neutralising agent must be a basic material in a form that is capable of reacting with the free uncombined acid present to convert the free acid to salt. Of these examples, those compounds with nutrient value are especially advantageous, and adding an aqueous solution is the most easily controllable manner of neutralising the free acid; it will therefore be seen that aqueous ammonia solution is the preferred neutralising agent.

Granulation may be carried out in any suitable granulator, for example, a rotary drum or a paddle mixer.

The mixture fed to the granulator may include material recycled from other stages of the process, or fertiliser materials, for example, potassium chloride, ammonium sulphate, potassium sulphate may also be added.

The drying of the granules may be carried out, for example, in a rotary dryer by means of a stream of hot air or other gas.

The invention further consists in a granular fertiliser when prepared by the method as described in any of the preceding nine paragraphs.

The invention will be further described with reference to the following examples:—

EXAMPLE 1:

A fertiliser was formulated from muriate of potash, granular diammonium phosphate of such size that 95% of the material lies in the range 1—4 mm. diameter, 20% superphosphate and 25% urea.

All of the free acid present was contained in the superphosphate constituent.

The free acid content of the monocalcium-phosphate-containing constituent (i.e. the superphosphate) was determined by the titration with standard alkali to pH 4,6 of a 2% aqueous extract of the monocalcium-phosphate-containing constituent. It is defined as the weight of P_2O_5 equivalent to the amount of standard alkali thus required, and expressed as the per cent by weight of P_2O_5 thus found based on the weight of the monocalcium-phosphate-containing constituent.

The mixed ingredients were granulated in a rotary tube conditioner with the addition of steam. Before this granulated material was passed to the dryer, the free acid content of the superphosphate was reduced to 1% by means of aqueous ammonia solution.

The product was substantially dried in a rotary dryer in a stream of hot air at 105° C and the dried product was subsequently cooled in a rotary cooler.

Two points which should be noted in this Example are that the granular diammonium phosphate, having a relatively small surface for unit weight, does not exert an appreciable neutralising effect in the time allowed for the process, and that an air temperature of 105° C in the dryer gives a mass temperature in the region of 80° C.

EXAMPLE 2:

A mixture composed of 70% urea and 30% superphosphate (the free acid P_2O_5 content of the latter being 3.76%) was granulated in a rotary drum using steam.

The mixture was then divided into two equal parts. One part was ammoniated by spraying with 30% aqueous ammonia solution so as to reduce the free acid P_2O_5 content down to 1%. Each part was then dried sepa-

rately under identical conditions in a rotary dryer for 20 minutes. The air stream temperature was 80° C and the final material temperature in both cases was 73° C.

In the case of the unammoniated sample a great deal of granule growth occurred during the drying period, forming large agglomerates, whereas the ammoniated portion showed little or no signs of growth during this period.

EXAMPLE 3:

A mixture composed of muriate of potash, sulphate of ammonia, single and triple superphosphate and 15% urea was granulated using steam to give a fertiliser containing 10% N, 10% P_2O_5 and 18% K_2O.

The granulate was divided into two equal portions and one of the portions was ammoniated with aqueous ammonia as in Example 2 so that the free acid P_2O_5 content of the single and triple superphosphates was reduced to 1%. The two portions were then dried separately in a co-current rotary dryer, the emergent material being at 90°C. The unammoniated granules quickly developed a sticky glistening surface and adhered to each other and the surfaces of the lifters in the dryer. However, the ammoniated portion showed no sticky character and was completely free-flowing throughout.

WHAT WE CLAIM IS:—

1. A method for reducing dryer stick in the preparation of granular fertilizers, which comprises carrying out the preparation in the three separate and consecutive steps (i) preparing a mixture containing monocalcium phosphate, urea, and free uncombined acid and granulating the mixture (ii) after granulation applying a neutralising agent to the formed granules to reduce the overall acid content and (iii) drying the granules.

2. A method as claimed in claim 1, in which the free acid is reduced to 1% (expressed as P_2O_5) by weight of the monocalcium phosphate-containing constituent present.

3. A method as claimed in claim 1, or 2, in which the mixture contains ordinary superphosphate or triple superphosphate.

4. A method as claimed in any one of claims 1 to 3, in which between 15% and 70% by weight of urea is present.

5. A method as claimed in any of claims 1 to 4, in which drying is carried out at temperatures less than 95° C.

6. A method as claimed in claim 5, in which drying is carried out at from 80° to 90° C.

7. A method as claimed in any one preceding claim, in which the neutralising agent has a nutrient value.

8. A method as claimed in claim 7, in which the neutralising agent is one or more of gaseous ammonia, aqueous ammonia solution or finely divided solid diammonium phosphate.

9. A method as claimed in any one preceding claim, in which the mixture fed to the granulator includes potassium chloride, ammonium sulphate or potassium sulphate.

10. A method as claimed in any one preceding claim, in which sulphuric and/or phosphoric acid is added before or during granulation and before addition of neutralising agent.

11. A method for the preparation of granular fertilisers substantially as hereinbefore described with reference to any one of the Examples given.

12. Granular fertilisers prepared according to the method as claimed in any one preceding claim.

MARKS & CLERK,
Chartered Patent Agents,
Agents for the Applicants.

Leamington Spa: Printed for Her Majesty's Stationery Office, by the Courier Press (Leamington) Ltd.—1967. Published by The Patent Office, 25 Southampton Buildings, London, W.C.2, from which copies may be obtained.